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Adhesion of orthodontic brackets to indirect laboratory processed resin composite as a function of surface conditioning methods and artificial aging

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Short title: *Adhesion of brackets to indirect resin composite*

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Abstract: This study compared the shear bond strength of orthodontic brackets to laboratory processed indirect resin composites (IRC) after different surface conditioning methods and aging. Specimens made of IRC (Gradia Indirect, GC) (thickness: 2 mm; diameter: 10 mm) (N=80) were randomly assigned to one of the following surface conditioning methods: C-Control: no treatment; AA-Air-abrasion (50 μ m Al₂O₃ particles); DB-Diamond bur and HF-Etching with hydrofluoric acid (9.6%). After adhesive primer application (Transbond XT), orthodontic brackets were bonded to the conditioned IRC specimens using adhesive resin (Transbond XT). Following, storage in artificial saliva for 24 h at 37°C, the specimens were thermocycled (x1000, 5-55°C). The IRC-bracket interface was loaded under shear in a Universal Testing Machine (0.5 mm/min). Failure types were classified using modified Adhesive Remnant Index criteria. Data were analyzed using two-way ANOVA and Tukey's HSD (α =0.05). Surface conditioning method did not significantly affect the bond strength results (p=0.2020) but aging significantly decreased the results (p=0.04). Interaction terms were not significant (p=0.775). In both non-aged and aged conditions, non-conditioned C group presented the lowest bond strength results (MPa) (p<0.05). In non-aged conditions, surface conditioning with DB (8.03±0.77) and HF (7.87±0.64) showed significantly higher bond strength results compared to those of other groups (p<0.05). Thermocycling significantly decreased the mean bond strength in all groups (2.24±0.36-6.21±0.59) (p<0.05). The incidence of Score 5 (all adhesive resin remaining on the specimen) was the highest in HF group without (80%) and with aging (80%) followed by DB (40, 70% respectively). C groups without and with aging showed exclusively Score 1 type (no adhesive resin on the specimen) of failures indicating the least reliable type of adhesion.

Keywords: Adhesion, aging, bond strength, indirect resin composite, orthodontic brackets, surface conditioning

Introduction

There is an increasing demand for orthodontic therapies associated with improved esthetic needs in adult population [1]. Due to increased age and impaired dentition, adult orthodontic patients often have restorations made of direct or indirect resin composite, amalgam or ceramics onto which brackets need to be bonded.

Direct bonding of orthodontic brackets to natural dentition was introduced in 1965 [2] presenting in vitro shear bond strength values for orthodontic adhesives between 6 to 8 MPa [3]. Although direct bonding is a well-known procedure, bonding orthodontic brackets to the direct restorative materials and all-ceramic restorations are still considered a challenge. Bonding orthodontic brackets to direct and/or indirect restorative materials such as amalgam [4], methacrylate-based restorative resin composite [5-8], and dental ceramics [9] require initial surface conditioning methods.

Recently introduced laboratory processed indirect resin composite resin (IRC) materials tempt to solve problems inherent to the dental ceramics. IRCs are advocated for a wide range of indications such as inlays, onlays, laminate veneers, metal-free crowns and short-span anterior bridges [10] with some advantages such as favourable optical properties, reparability, fast and simple laboratory procedures [11]. IRCs contain higher density of inorganic ceramic fillers than traditional direct and indirect resin composites [12]. Moreover, the post-polymerization process for IRCs result in superior flexural strength to feldspathic porcelain, minimal polymerization shrinkage, and wear rates comparable to that of tooth enamel [13].

Adhesion of orthodontic brackets to natural dentition could withstand the orthodontic forces by large but their adhesion to IRCs may lead to debonding type of failures and cause discomfort and additional costs during the orthodontic therapy [14,15]. Although shear bond strength of metal orthodontic brackets to amalgam, methacrylate-based resin composites and dental ceramics have been widely investigated [14-17], their adhesion to IRCs have not been reported.

The objectives of this study therefore, were to investigate the effect of different surface conditioning methods on bond strength of metal orthodontic brackets to IRCs with and without aging. The null hypotheses tested were that a) surface conditioning methods and b) aging through thermal cycling would not show significant difference between groups in terms of bond strength.

Materials and Methods

Specimen preparation

IRC specimens (N=80) (Shade E1, Gradia Indirect, GC Corporation, Tokyo, Japan) (thickness: 2 mm; diameter: 10 mm) were fabricated using polytetrafluoroethylene (PTFE) moulds. Unpolymerized IRC was carefully condensed with a plastic instrument ensuring no air entrapment. The specimens were initially photo-polymerized (GC Steplight SL-I pre-curing light unit, GC Corporation) for 10 s from a distance of 2 mm. The specimen surfaces were coated with glycerin gel (GC Gradia Air Barrier, GC Corporation) in order to eliminate oxygen-inhibited layer and to ensure complete polymerization. Then the specimens were placed in laboratory photo-polymerization device (GC Laborlight LV-I, GC Corporation) for effective final polymerization for 5 minutes. After final polymerization, glycerin gel was removed under copious water.

Following photo-polymerization, the specimens were ground finished using #400, #600, #800, # 1200 and #2000 waterproof silicon carbide abrasive papers, respectively. Then the specimens were embedded in polymethylmethacrylate blocks with their polished bonding surfaces exposed. The blocks were ultrasonically cleaned in distilled water (Eurosonic Energy, Euronda SpA, Vicenza, Italy) for 10 minutes and dried with oil-free air for 30 s. One operator (E.Y.) prepared the specimens according to the manufacturer's recommendations. The specimens were divided into 4 subgroups (n=20 per group) to be conditioned with one of the following methods:

Surface conditioning methods

Group C: The non-conditioned IRC specimens acted as the control group.

Group AA: The specimen surfaces were air-abraded with 50 μm Al_2O_3 particles with a microetcher (Airsonic Mini Sandblaster, Hager & Werken, Duisburg, Germany) for 10 s from a distance of approximately 10 mm at 250 kPa.

Group DB: Specimens were roughening with a diamond bur (A-diamant, No:140-014, Acurata GmbH & Co. KG, Thurmansbang, Germany) using a high-speed hand piece under water-cooling.

Group HF: In this group, specimens were etched with 9.6 % hydrofluoric acid (Ultradent Porcelain Etch, Ultradent Products, Inc. Utah, USA) for 90 s, rinsed with distilled water for 60 s and dried with oil-free air for 30 s.

Adhesion procedures

A thin layer of adhesive primer (Transbond XT, 3M Unitek, Monrovia, California, USA) was applied on the conditioned specimen surface. Then adhesive paste (Transbond XT, 3M Unitek) was applied to the mesh base of the lower incisor metal bracket (Gemini, 3M Unitek; slot 0.018 inch) and then bracket was placed onto the specimen surface using a bracket plier. During the bonding, a constant load of 5 N was applied to the top surface of the bracket in order to obtain a uniform adhesive layer thickness. Excess adhesive paste was removed from the periphery of the bracket base using a dental explorer. The adhesive paste cement was photo-polymerized with a light emitting diode polymerization device (LED Elipar Freelight 2, 3M ESPE, St. Paul, USA) for 40 s to achieve adequate polymerization from two directions from a constant distance of 1 mm. All bonding procedures were performed by the same operator (G.S-T.) according to the manufacturer`s instructions.

Aging and bond strength test

The specimens were stored in distilled water at 37°C for 1 week. Half of the specimens from each group were randomly (n=10 per group) selected and subjected to thermocycling (Nova, Nova Co, Konya, Turkey) for 1000 cycles [9], between 5-55°C, with a transfer time of 30 s and a dwell time of 30 s [16]. After thermocycling, the specimens were loaded at the bracket-specimen interface at a crosshead speed of 0.5

mm/min in a Universal Testing Machine (Lloyd Instruments, Hampshire, UK) until failure. The bond strength was expressed in megapascals, derived from dividing the load at failure (N) by the bonding area (mm^2). The bracket surface area according to the manufacturer was verified under a stereo-microscope (9.81mm^2 , 0.018 inch slot size).

Failure analysis

Debonded specimen surfaces were examined under a stereomicroscope (Leica MZ 12, Leica Microsystems, Bensheim, Germany) at x80 magnification. Failure types were scored according to the modified adhesive remnant index (ARI) as follows: 1= no adhesive resin, 2= less than 10 per cent of the adhesive resin, 3= more than 10 per cent but less than 90 per cent of adhesive resin, 4= more than 90 per cent of adhesive resin, and 5= all adhesive resin remaining on the specimen [17].

Statistical analysis

The sample size was calculated using a power analysis software (G*Power Version 3.1.9, Dusseldorf, Germany) considering α equal to 5%, effect size equal to 0.60 and power of 80% according to 1-Way ANOVA test. Based on the calculations, 9 specimens per group yielded to 86% power but 10 specimens were employed in the study.

Data (MPa) were analyzed using a statistical software package (SPSS Software V.22, Chicago, IL, USA). Kolmogorov-Smirnov and Shapiro-Wilk tests were used to test normal distribution of the data. As the data were normally distributed, 2-way analysis of variance (ANOVA) and Tukey's HSD tests were applied to analyze the data where bond strength was the dependent variable and surface conditioning methods (4 levels), and aging (2 levels: dry versus thermocycling) as independent variables. $P < 0.05$ was considered to be statistically significant in all tests.

Results

While surface conditioning method did not significantly affect the bond strength results ($p=0.2020$), aging through thermocycling significantly decreased the results ($p=0.04$) (Table 1). Interaction terms were not significant ($p=0.775$).

In both non-aged and aged condition non-conditioned C group presented the lowest bond strength results (MPa) ($p<0.05$) (Table 2). In non-aged conditions, surface conditioning with DB (8.03 ± 0.77) and HF (7.87 ± 0.64) showed significantly higher bond strength results compared to those of other groups ($p<0.05$). Thermocycling significantly decreased the mean bond strength in all groups (2.24 ± 0.36 - 6.21 ± 0.59) ($p<0.05$).

The incidence of Score 5 was the highest in HF group without (80%) and with aging (80%) followed by DB (40, 70% respectively). C groups without and with aging showed exclusively Score 1 type of failures indicating the least reliable type of adhesion (Table 3).

Discussion

This study was undertaken in order to suggest the most effective surface conditioning method for bonding orthodontic metal brackets on the laboratory processed IRCs. Based on the results of this study, since surface conditioning methods did not affect the adhesion results significantly, the first null hypothesis could be accepted. After aging, the results decreased significantly in all groups, yielding to rejection of the second hypothesis.

Typically, shear bond strength of 6 to 8 MPa is considered clinically adequate for bonding orthodontic brackets to teeth [3]. The mean shear bond strengths of orthodontic brackets to the IRC material in this study after different surface conditioning methods fell within this range and therefore could be considered sufficient for clinical applications. In one previous study, tensile strengths for metal brackets bonded to microfilled and hybrid resin composite surfaces indicated 7.8 MPa [18] and in another one bond strength of ceramic brackets to resin composite ranged 17.1 between 19.2 MPa [7]. Newman et al. [19] examined the bonding of steel brackets to resin composite surfaces using a two-paste composite adhesive and

subjected to thermocycling, reporting mean bond strength of 9 MPa. These studies also clearly indicate that metal brackets cannot transmit light compared to ceramic ones and therefore result in lower bond strength on resin composites.

In order to improve the composite-composite adhesion, several conditioning methods have been suggested [20-22]. Such methods aim for improving the adhesion both on the inorganic filler and the cross-linked matrix of the resin composite. The common clinical trend for roughening the surface with a bur, green stone or by air-abrasion adds mechanical retention to the orthodontic substrates prior to bracket bonding [23]. The studies in the field of restorative dentistry indicated that preparing the resin composite surface prior to repair usually results in a increase in bond strength with the use of a bur [24], rotary abrasive stone [25] or an abrasive wheel [26].

In this study, in non-aged conditions, surface conditioning with DB (8.03 ± 0.77) and HF (7.87 ± 0.64) showed significantly higher bond strength results compared to those of the other groups. HF is a strong acid that dissolves the inorganic fillers of resin composite, creating porosity that serves for micro-mechanical retention [27]. In fact, both air-abrasion with Al_2O_3 particles and surface roughening with diamond burs, remove the superficial resin matrix and exposes the fillers of resin composite whereas HF literally removes the fillers from matrix. However, the type of micro-mechanical retention created with these two methods, are mainly on the surface. In this study, the IRCs were freshly prepared but in clinical conditions, resin composites may undergo aging and available free radicals on the surface may decrease over time. Nevertheless, surface conditioning with sodium bicarbonate, Al_2O_3 particle abrasion, HF or roughening with diamond burs were reported to deliver clinically acceptable shear bond strength values also on aged resin composite [23,28].

Thermocycling of specimens simulates the effects of thermally-induced stresses within the oral cavity [29]. The results indicated the aging effect on the IRC-bracket interface with decreased bond results. Although a dramatic decrease was not experienced, prolonged duration of aging may decrease the results further. Aging aspect has received less attention in the orthodontic literature, providing that the

nature of bonding is semi-permanent in orthodontics [29]. Thus, the duration of thermocycling in this study could be considered clinically relevant at least for orthodontic applications.

When a bracket bonded with resin adhesive is removed from the substrate surface, failure could occur at one of three interfaces: between the adhesive and the bracket, within the adhesive itself or between the adhesive and the substrate surface. Debonding procedures must be accomplished with a minimal or no risk of iatrogenic damage to the restoration surface after bracket removal [9]. In order to obtain a smooth and glossy restoration surface, residual adhesives must be carefully removed and polished with finishing-polishing kits so that discoloration and plaque accumulation could be prevented. The failure site is highly influenced by the bond strength between the substrate and the adhesive resin, the strength of the bond between the adhesive resin and the bracket base, and the mechanical properties of the adhesive resin [30]. In the present study, the highest amount of residual adhesives left on the IRC after HF conditioning. This indicates a strong bond between the IRC surface and the adhesive resin that exceeded the adhesive strength between the adhesive resin and the bracket surface. This type of failure will ensure reliable adhesion to the IRC but may result in debonding of the bracket from the resin surface. On the other hand, after AA and DB conditioning, frequency of Scores 3 and 4 indicated that adhesive resin was partially left on the IRC and also on the bracket base. This type of failure would lead to less damage on the IRC during removal of the resin after bracket debonding but also ensure sufficient adhesion between the adhesive resin and the bracket base.

From the clinical perspective, since HF has been considered a hazardous compound, and air-abrasion methods require additional devices, the use of diamond burs could be considered more practical. Yet, their effect on aged IRCs need to be verified in future studies.

Conclusions

From this study, the following could be concluded:

1. Adhesive strength of metal orthodontic brackets to laboratory processed indirect resin composite was not acceptable without surface conditioning the substrate, when 5 MPa was considered as clinically acceptable value.
2. Conditioning the indirect resin composite surface either with air-abrasion, diamond bur or 9.6% hydrofluoric acid increased the adhesion of brackets to the substrate but aging through 1000 thermocycling decreased the results in all groups.
3. Failure types were predominantly between bracket-adhesive resin interfaces after all surface conditioning methods whereas in the control group it was exclusively between the substrate and adhesive resin.

Clinical Relevance

Adhesion of metal brackets to laboratory processed indirect resin composite could be improved when the substrate is conditioned with either air-abrasion (50 micron Al_2O_3), diamond bur or 9.6% hydrofluoric acid. Considering the adhesion results and failure types, diamond bur may be considered the most feasible method for practitioners, as it does not require additional armamentarium for air-abrasion and eliminate the use of hazardous HF in the mouth.

Conflict of interest

The authors did not have any commercial interest in any of the materials used in this study.

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Captions to tables:

Tables:

Table 1. Effect of surface conditioning and aging methods on the bond strengths of brackets to indirect resin composite based on 2-way ANOVA and Tukey`s test.

Table 2. Mean±standard deviation (SD) of shear bond strength values and standard deviations (SD) for each group according to surface conditioning and aging parameters. C: Control; AA: Air-abrasion; DB: Dental bur; HF: Hydrofluoric acid. Different upper-case letters in each column for each condition indicates significant differences ($p<0.05$).

Table 3. Frequency (%) of failure types in each experimental group according to modified adhesive remnant index (ARI) scores (1= no adhesive resin, 2= less than 10 per cent of the adhesive resin, 3= more than 10 per cent but less than 90 per cent of adhesive resin, 4= more than 90 per cent of adhesive resin, and 5= all adhesive resin remaining on the specimen). TC: Thermocycling; NTC: No thermocycling.

Tables:

	Bond Strength (MPa)	
	F	p
Surface Conditioning Method	1.658	0.202
Aging	4.371	0.04*
Surface Conditioning*Aging	0.082	0.775

Table 1. Effect of surface conditioning and aging methods on the bond strengths of brackets to indirect resin composite based on 2-way ANOVA and Tukey`s test.

Surface Conditioning Method	Bond Strength (MPa)		p
	Aged	Non-Aged	
	Mean±SD	Mean±SD	
C	2.24±0.36 ^a	3.59±0.27 ^a	0.001
AA	4.24±0.49 ^b	6.19±0.59 ^b	0.001
DB	6.21±0.59 ^c	8.03±0.77 ^c	0.001
HF	5.12±0.52 ^d	7.87±0.64 ^c	0.001

Table 2. Mean±standard deviation (SD) of shear bond strength values and standard deviations (SD) for each group according to surface conditioning and aging parameters. C: Control; AA: Air-abrasion; DB: Dental bur; HF: Hydrofluoric acid. Different upper-case letters in each column for each condition indicates significant differences (p<0.05).

Experimental Groups	Score 1	Score 2	Score 3	Score 4	Score 5
C-TC	100	0	0	0	0
C-NTC	100	0	0	0	0
AA-TC	10	0	0	60	30
AA-NTC	0	20	10	50	20
DB-TC	0	0	0	30	70
DB-NTC	0	0	10	50	40
HF-TC	0	0	0	20	80
HF-NTC	0	0	0	20	80

Table 3. Frequency (%) of failure types in each experimental group according to modified adhesive remnant index (ARI) scores (1= no adhesive resin, 2= less than 10 per cent of the adhesive resin, 3= more than 10 per cent but less than 90 per cent of adhesive resin, 4= more than 90 per cent of adhesive resin, and 5= all adhesive resin remaining on the specimen). TC: Thermocycling; NTC: No thermocycling.